A

Project Report

On

Steady state simulation of Extractive Distillation system using Aspen Plus

Submitted by

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Under the guidance of

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CERTIFICATE

This is certified that the work contained in the thesis entitled **"Steady state simulation of Extractive Distillation System using Aspen Plus"** *submitted by* ***Pritam Kumar Bala (111CH0595)****, has been carried out under my supervision and this work has not been submitted elsewhere for a degree.*

_____

Date:

Place:

(Thesis Supervisor)

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ABSTRACT

In this project work, we report Aspen-plus simulation results of an azeotropic system *viz*. isopropanol-water which forms a minimum boiling azeotrope. Because of its large scale industrial application, separation of isopropanol-water mixture using the distillation process gained prominence. In this work, two routes were followed leading to the simulation. The first involved more conventional extractive distillation mechanism where Dimethyl sulfoxide (DMSO) was used as an entrainer. A percentage purity of 99.9% was achieved w.r.t isopropanol in the distillate or top product. However, a significant percentage of DMSO remained in the bottom product. Since the recovery of DMSO was critical, another auxiliary column was configured with the main scheme and a recovery of more than 99% was achieved as the bottom product from the second column. Since this process involved two distillation columns, a relatively more contemporary and modern distillation mechanism was resorted to *viz*. divided wall distillation process. Improvisations were made in the flow sheet to mimic divided wall distillation mechanism as Aspen-plus don't have such specific simulator. The results were comparable to what was achieved in extractive distillation process.

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NOTATIONS AND ABBREVIATIONS

CPI = Chemical process Industries

DMSO = Dimethylsulfoxide

DWC = Divided wall distillation column

THF = Tetrahydrofuran

FE1 = Entrainer inlet stream

F1 = Feed inlet stream

B1 = Bottom product stream

D1 = Top product stream

ROM = Read only memory

IC = Integrated circuits

CD = Compact disk

LCD = Liquid crystal display

CPU = Central processing units

IPA = Isopropyl alcohol

NRTL = Non-random two liquid

LAB = Linear Alkyl Benzene

F = feed in DWC

B = Bottom product in DWC

D = Top product in DWC

S3 = Mid product from section 2 column

LNS2 = liquid product returning from section 2 column to stripping column in DWC

VNS1 = Vapor returning to enriching section from section 2 column in DWC

v = Vapor going into section 2 column in DWC

l = Liquid going into section 2 column in DWC

$\tau^{12}$and τ^{21} = dimensionless interaction parameter

Δg^{12} and Δg^{21}= interaction energy parameters

R = gas constant

T= Absolute Temperature

U_{ij} = Energy between surfaces i and j

CHAPTER 1

INTRODUCTION

In this chapter we have discussed the fundamentals of distillation in general and extractive distillation in particular. To separate azeotropic compositions formed between various components constituting mixtures have been an area of research for several decades and literatures on that are highlighted here. The background and the motivation behind carrying out the study are summarized along with the specific objectives.

1.1 Prelude

Distillation is one of the most fundamental and sought after techniques prevalent in various process industries. The success of distillation as a unit operation depends on the fundamental factor called relative volatility. The higher the relative volatility, the easier the separation would be. However, there arise many situations when the relative volatility between the constituents of the mixture is quite adjacent to each other and hence the degree of difficulty to separate them increases.

Azeotropes are constant boiling mixtures when vapour and liquid phase compositions are equal and no separation by conventional distillation is possible beyond that point. The greater the non-ideality in the mixture, the more probable is the chance of an azeotrope formation. Ideal solutions obey Raoult's law and non-ideal solutions deviate from that. There are two possibilities: positive deviation or negative deviation. Generally, this deviation occurs large because of the dissimilarity in the chemical configuration of the constituting species. This differences lead to non-uniformity in the inter-molecular forces of attraction between them. In positive from deviation, the partial predicted by the Raoult's law is less than the practical scenario, meaning higher escaping tendencies between the constituting molecules, leading to a minimum boiling azeotrope, whereas, in case of negative deviation, the escaping tendencies are less for the molecules, leading to a maximum boiling azeotrope [1-2]. The occurrences of minimum boiling azeotrpes are more common. Isopropanol-water system forms a minimum boiling azeotropic system.

1.2 Application

Various methods have been developed and used industrially to separate azeotropic mixtures. The methods are highlighted below:

- Pressure swing distillation process
- Azeotropic distillation with a light entrainer
- Extractive distillation with a heavy entrainer (solvent)
- Pervaportion

Some very popular and commonly occurring azeotropic mixtures are: isopropanol-water, ethanol-water, tetrahydrofuran-water, n-butanol-water, acetone-methanol, isopentane-methanol, acetic acid-water and acetone-chloroform from a wide list [2].

1.3 Uses of Isopropanol

Isopropyl alcohol has wide application. It is used as a solvent, as a chemical intermediate, in medical field, in automotive and as anaesthesia. Major fraction of IPA is used as solvent and in industrial applications. A tiny fraction is consumed for household use and in personal care products. IPA is one of the major chemical in pharmaceutical industries due to its nature of forming low toxicity of any residues. Few fractions are used as intermediate in various industrial applications. Acetone is a gasoline additive, is one of the intermediate formed from IPA.

Solvent

One of the important characteristics of IPA is that it can dissolve wide range of non-polar products. Isopropanol few characteristics like its nontoxic and it evaporates quickly leaving no residue makes it an ideal product to use as compared to other solvents like ethanol. Hence major fractions of IPA produced are used as solvent. It is also used to clean delicate electronic devices like ROM cartridges, CD, DVD, LCD screen. It is also used to remove stains and removing paint.

Intermediate

Isopropyl alcohol is used in several methods as intermediate:-

- It is used in esterification process to produce derivatives of isopropyl acetic acids which are again used as solvents.
- A herbicide and an ore floatation agent, sodium isopropylxanthate, is produced by reacting IPA with sodium hydroxide and carbon disulphide.
- Titanium isopropoxide is formed when IPA reacts with Titanium tetrachloride.
- Aluminium isopropoxide is produced when IPA reacts with aluminium.

Medical

IPA is widely used as sanitizing cushions having 60–70% arrangement of IPA in water. It is also utilized as a hand sanitizer and as a water-drying guide for the antipathy of otitis externa. This is also known as swimming ear.

Early uses as an anaesthetic

Previously Isopropyl alcohol was used as anaesthesia. But due to its numerous negative disadvantages its use as anaesthesia was stopped. IPA can be utilized as a dissolvable or as a sedative by breathing in the vapour or orally. Its use was soon stopped due to various side effects including respiratory disturbance, inner drying, and visual and auditory issues.

Automotive

IPA is a critical altering in "gas dryer" fuel included substances. In discriminating sums, water is an issue in fuel tanks, as it partitions from the gas, and can harden in the supply lines at chilly temperatures. Alcohol does not remove water from fuel; rather, the alcohol solubilizes water in gas. At the point when dissolvable, water does not speak to the same danger as insoluble water, as it will no more gather in the supply lines and stop. Isopropyl liquor is habitually sold in vaporized jugs as a win shield de-icer. IPA is also used to oust brake fluid takes after from water controlled halting computerizations, leads to poor braking.

1.4 Simulation multiplicity

Most of the equations involved in designing distillation columns *viz*. component mass balances, energy balances, vapour-liquid phase equilibrium relationships, column hydraulic equations are non-linear. Since non-linear equations have multiple roots, convergence of solution is always going to be difficult. It means that we may have multiple results for the

same set of inputs also known as 'output multiplicity' [2]. This adds to the already existing complexity in simulating distillation columns. Therefore, in Aspen-plus simulation methodology, this particular aspect of convergence is always going to be a key issue.

A simple schematic of extractive distillation simulation configuration is shown below:

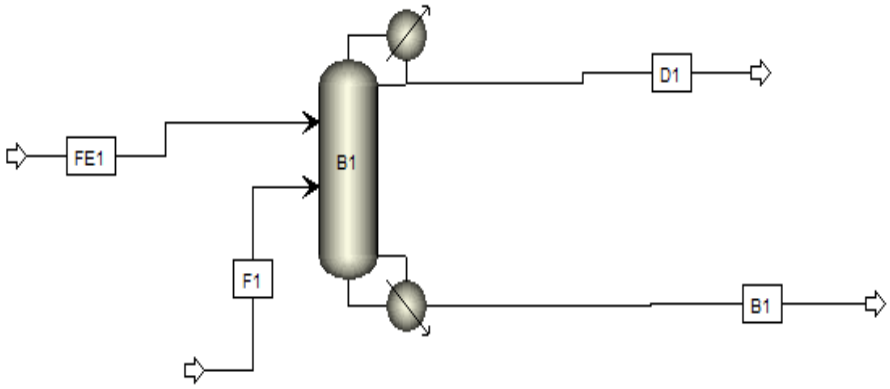

Fig-1.1 Schematic of an Extractive Distillation process

FE1 is entrainer feed. F1 is fresh feed of azeotrope. B1 is bottom product and D1 is distillate. Further discussion will be carried out later.

1.5 Research Objectives

- To study the functioning of anextractive distillation column in steady state with special emphasis on a minimum boiling azeotropic mixture (**isopropanol-water)** where isopropanol is extracted using a suitable entrainer *viz.* **dimethyl sulfoxide (DMSO).**
- To study the optimization of the number of trays, feed plate, entrainer plate and purity of distillate obtained by varying the process variables like reflux ratio and distillate rate.
- To study the recovery of entrainer from water using another distillation column or extracting all the three in a single distillation column with special emphasis on a **divided wall distillation system.**

CHAPTER-2

LITERATURE REVIEW

A distillation column has two sections namely stripping section and rectifying section. In order to separate three component mixture into pure products two distillation columns are required. So in total four sections are required to separate three component mixture [3]. Each section of the column namely stripping sections and rectifying section has a reboiler and a condenser respectively. Getting output with minimum use of energy has been a new trend in production line. This demand is achieved by dividing wall distillation column (DWC). Separation of three components is done in single column rather than in two columns as done in conventional method. Major advantage in DWC is by avoiding remixing problem which was quite an issue in conventional distillation methods. Hence DWC is an energy efficient method and an alternative to conventional method [4]. In DWC a wall in between the column separates the product zone and side stream zone and increase the efficiency of DWC as compared to the conventional one [5]. Dividing wall inside the column plays a crucial role in product purity. In order to have high purity of product insulation of this wall is properly done to avoid heat transfer through it. In general dividing wall is placed in the middle but the position can be varied in accordance to medium boiling component. This change in placing the wall is seen when medium boiling component is small as compared to overhead and bottom product obtained [6]. Few assumptions were taken before DWC model was prepared *viz.* pressure maintained in column is constant with no dynamic vapor flow, energy balance and change in enthalpy is neglected. Various control strategies were compared using this model [7]. Another assumption is for ideal components where mass and heat transfer takes place between its liquid and vapor phases [8].

In this paper minimum boiling azeotrope (Isopropanol- water) is separated using an entrainer DMSO using both conventional method of distillation and dividing wall distillation column (DWC). Aspen plus software provides various simulation models so that optimum input variables can be calculated before starting a process in any production plant. In Aspen plus

V8.4 divided wall distillation column is not provided hence a mimic model has been taken to get an optimum result which is exact replica of dividing wall distillation column in terms of functioning.

2.1 Extractive Distillation

Separation of azeotropic mixture had been a major challenge because after specific concentration it forms constant boiling mixture hence further purification was not possible. This problem was addressed by extractive distillation. This method used a third component which combines with one of the azeotropic component and increases the relative volatility between azeotropic components. The third component is a miscible, non-volatile solvent called entrainer. More volatile component is obtained as top product and less volatile component along with entrainer is obtained at the bottom. Further separation of entrainer and another component obtained as bottom product is achieved by another auxiliary column.

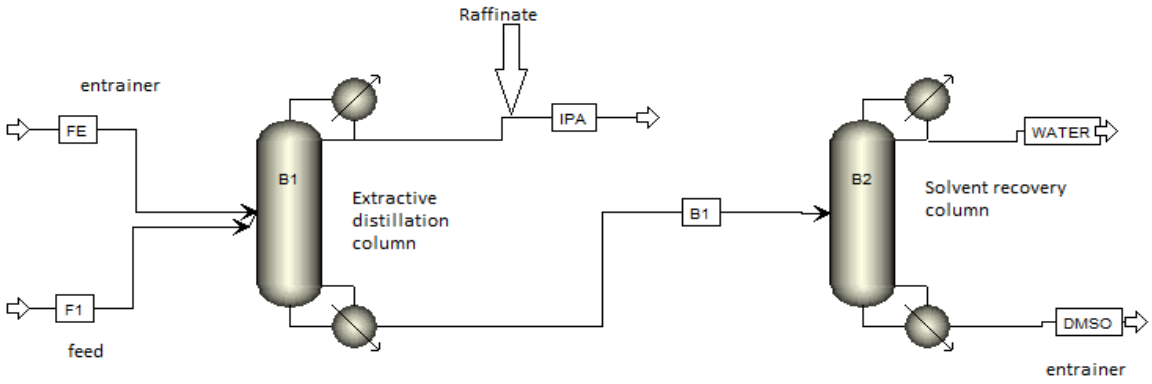

Fig-2.1 Configuration of extractive distillation

2.1.1 Types of extractive distillation

Extractive distillation process can be split into three important categories:

- Separation of minimum boiling azeotropes
- Separation of maximum boiling azeotropes
- The separation of low relative volatility non-azeotropic mixtures.
- For separating binary mixtures into pure components.

In extractive distillation, importantly, a solvent is purposely added two widen the boiling point differences of the constituting species of the mixture or to avoid forming any azeotropes. So selection of a proper solvent is always remained very important in the overall success of the process.

2.2 Choice of Solvent

A solvent to be used as an entrainer, preferably, should have the following salient features or characteristics:

•Relative volatility of the key component should be enhanced.
•Ratio of solvent to non-solvent should be quite less.
• Easily soluble with feed components and do not form any two phase with it.
•Easily separable from the bottom product.
•Inexpensive and readily available.
•Stable at the operating temperature of the distillation column.
•Non-reactive with the components in the feed mixture.
•Low latent heats.
•Non-corrosive and non-toxic.

CHAPTER 3

STEADY STATE SIMULATION

(Results and Discussion)

3.1 Simulation performed [2]

In this section we use an extractive distillation column as an example to demonstrate how to build a steady-state simulation. Aspen V8.4 was used to simulate this model. This is one column of an overall two-column system for separating isopropanol and water. The column configuration is shown in Figure 3.1. There are two feed streams entering this column: a fresh feed (mixture of IPA and water) is fed in 35thStage, and a solvent entrainer stream (dimethyl sulfoxide) is fed in 7th Stage. The column has total of 41 stages (including total condenser and reboiler). Other operating conditions are shown in figure 3.1.

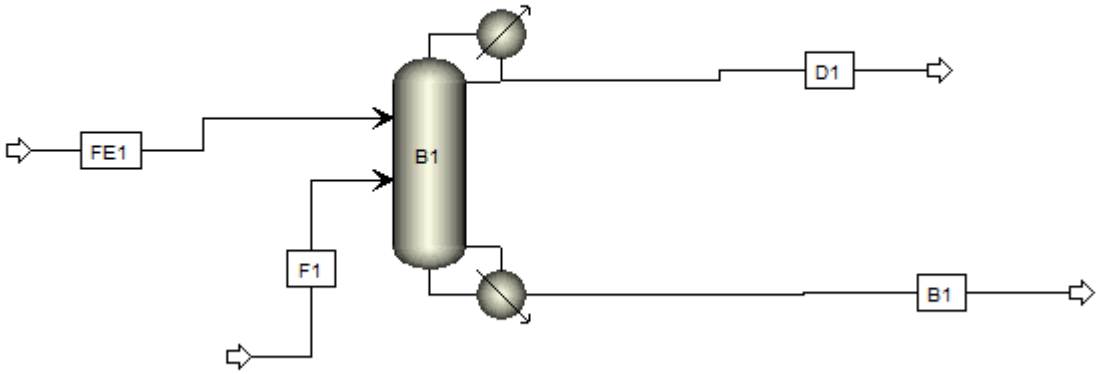

Fig 3.1. Schematic representation of the extractive distillation system

Conditions:

Entrainer(FE1):-[DMSO]

Temperature- 72^{o}C

Pressure- 1.1 atm

Flow rate- 100kmol/hr

Feed(F1):- [isopropanol + water]

Temperature- 25^{o}C

Pressure- 1.3 atm

Flow- 100 kmol/hr Mole ratio – 0.5(isopropanol) and 0.5(water)

In this setup shown above FE1 stream is given in the 7th stage and F1 in 35th stage. Column B1 is divided into three sections namely enriching section, extraction section, stripping section. FE1 stream has DMSO solvent which is an entrainer. It's miscible, non-volatile and has high boiling point of 194.4^{o}C. It increase the relative volatility of isopropanol water mixture and separates water from isopropanol and takes water with it down the column to stripping section which is below the feed stream (F1) section. Isopropanol being more volatile moves up the column to enriching section which is above the entrainer feed stream. Isopropanol is obtained from top and mixture of water and entrainer is obtained as bottom product. Bottom product is further separated in another auxiliary column separating all three components and utilizing entrainer back in the system as recycle to entrainer stream.

Three components ISOPROPYL ALCOHOL, WATER and DMSO were taken. Base method was **NRTL** (Non-random two liquid) model. This model is recommended for highly non ideal chemical systems and can be used for both vapour-liquid and liquid-liquid equilibrium applications. The equation for the NRTL model is:

$$\begin{cases} \ln\ \gamma_1 = x_2^2 \left[\tau_{21} \left(\frac{G_{21}}{x_1 + x_2 G_{21}} \right)^2 + \frac{\tau_{12} G_{12}}{(x_2 + x_1 G_{12})^2} \right] \\ \ln\ \gamma_2 = x_1^2 \left[\tau_{12} \left(\frac{G_{12}}{x_2 + x_1 G_{12}} \right)^2 + \frac{\tau_{21} G_{21}}{(x_1 + x_2 G_{21})^2} \right] \end{cases}$$

With

$$\begin{cases} \ln\ G_{12} = -\alpha_{12}\ \tau_{12} \\ \ln\ G_{21} = -\alpha_{21}\ \tau_{21} \end{cases}$$

In here τ_{12} and τ_{21} are the dimensionless interaction parameters, which are related to the interaction energy parameters Δg_{12} and Δg_{21} by:

$$\begin{cases} \tau_{12} = \frac{\Delta g_{12}}{RT} = \frac{U_{12}-U_{22}}{RT} \\ \tau_{21} = \frac{\Delta g_{21}}{RT} = \frac{U_{21}-U_{11}}{RT} \end{cases}$$

R is the gas constant and T, absolute temperature. U_{ij} is the energy between molecular surfaces i and j. U_{ii} is the energy of evaporation where U_{ij} has to be equal to U_{ji}, however, Δg_{ij} is not necessary equal to Δg_{ji}.

Since this mixture is highly non-ideal NRTL method is preferred.

Aspen-plus v 8.4 software was used for simulation work. The preferred column was Radfrac simulator with 39 trays (excluding condenser with reflux drum and reboiler). Condenser was a total condenser and reboiler was kettle type. Valid phases were vapor-liquid. Convergence method was opted for strongly non-ideal liquid. Distillate rate was taken to be 50 kmol/hr and reflux ratio as 0.5, as initial guesses.

Feed plate location was taken on 35th stage and entrainer plate on 7th stage (counted from top). Condenser pressure was taken as 1 atm with stage pressure drop of *ca*. 0.0068 atm. When simulated to the given condition the following results were obtained:

Table-3.1Stream table of extractive distillation column

final year pro					
Stream ID		B1	D1	F1	FE
From		B1	B1		
To				B1	B1
Phase		LIQUID	LIQUID	LIQUID	LIQUID
Substream: MIXED					
Mole Flow	kmol/sec				
ISOPR-01		3.20967E-6	.0138856	.0138888	0.0
WATER		.0138856	3.20203E-6	.0138888	0.0
DIMET-01		.0277777	7.64048E-9	0.0	.0277777
Mole Frac					
ISOPR-01		7.70320E-5	.9997689	.5000000	0.0
WATER		.3332565	2.30546E-4	.5000000	0.0
DIMET-01		.6666665	5.50115E-7	0.0	1.000000
Total Flow	kmol/sec	.0416666	.0138888	.0277777	.0277777
Total Flow	kg/sec	2.420765	.8345310	1.084878	2.170418
Total Flow	cum/sec	2.58774E-3	1.15352E-3	1.29244E-3	2.07118E-3
Temperature	K	424.5966	355.4929	298.1500	345.1500
Pressure	N/sqm	1.28885E+5	1.01325E+5	1.31723E+5	1.11458E+5
Vapor Frac		0.0	0.0	0.0	0.0
Liquid Frac		1.000000	1.000000	1.000000	1.000000
Solid Frac		0.0	0.0	0.0	0.0
Enthalpy	J/kmol	-2.1604E+8	-3.0653E+8	-3.0210E+8	-1.9707E+8
Enthalpy	J/kg	-3.7185E+6	-5.1014E+6	-7.7351E+6	-2.5222E+6
Enthalpy	Watt	-9.0015E+6	-4.2573E+6	-8.3916E+6	-5.4743E+6
Entropy	J/kmol-K	-2.3891E+5	-4.2653E+5	-3.1015E+5	-3.2964E+5
Entropy	J/kg-K	-4112.137	-7098.666	-7941.350	-4218.865
Density	kmol/cum	16.10158	12.04049	21.49249	13.41157
Density	kg/cum	935.4755	723.4674	839.4020	1047.914
Average MW		58.09835	60.08623	39.05560	78.13504
Liq Vol 60F	cum/sec	2.23080E-3	1.05982E-3	1.31070E-3	1.97992E-3

Here we are getting mole fraction of isopropanol 0.9997689. In order to increase the purity we used design specs and vary function.

3.2 Analysis

In design specs option type taken as mole purity with target as 0.9999999 with component as isopropanol and stream is DI. In vary option type taken as distillate rate with lower bound 0.01 kmol/sec and upper bound as 0.02 kmol/sec then simulation run which completed in 18 iterations. Taking second design spec as mole ratio of IPA and IPA+ water as 0.001 from bottom (B1). Vary taken as reflux ratio ranging 0.1 to 1.0. It converges in 16 iterations. Reflux ratio came 0.6895 and distillate flow rate as 50.007 kmol/hr. The results we are getting are quite encouraging as we are getting a purity of 0.9999 isopropanol from top as distillate.

In the temperature profile plot given below there is a sharp rise near 7^{th} plate. This change is observed as the entrainer is given into the column at this stage which is at higher temperature as compared to column temperature. Then there is a general rise in curve as temperature down the column increases due to the presence of reboiler. But sharp fall near the 35^{th} plate is observed because feed which is at 298.15 K in given into column which brings down the temperature of the column. After 35^{th} column general rise in curve is observed.

In liquid composition profile IPA mole fraction goes on increasing to the top of the column as its more volatile as compared to other two components. Sharp rise is observed after 7^{th} plate as DMSO (entrainer) is introduced. Mole fraction of IPA is nearly 0 at 41^{st} stage. Likewise water mole fraction increases after 35^{th} plate as feed first comes in contact with DMSO there and DMSO separates water from feed.

Table-3.2Stream table of extractive distillation column using design specs and vary

final year pro					
Stream ID		B1	D1	F1	FE
From		B1	B1		
To				B1	B1
Phase		LIQUID	LIQUID	LIQUID	LIQUID
Substream: MIXED					
Mole Flow	kmol/sec				
ISOPR-01		1.38889E-5	.0138750	.0138888	0.0
WATER		.0138750	1.38762E-5	.0138888	0.0
DIMET-01		.0277777	1.27465E-8	0.0	.0277777
Mole Frac					
ISOPR-01		3.33334E-4	.9990000	.5000000	0.0
WATER		.3330003	9.99084E-4	.5000000	0.0
DIMET-01		.6666664	9.17751E-7	0.0	1.000000
Total Flow	kmol/sec	.0416666	.0138888	.0277777	.0277777
Total Flow	kg/sec	2.421214	.8340819	1.084878	2.170418
Total Flow	cum/sec	2.58811E-3	1.15280E-3	1.29244E-3	2.07118E-3
Temperature	K	424.4920	355.4828	298.1500	345.1500
Pressure	N/sqm	1.28885E+5	1.01325E+5	1.31723E+5	1.11458E+5
Vapor Frac		0.0	0.0	0.0	0.0
Liquid Frac		1.000000	1.000000	1.000000	1.000000
Solid Frac		0.0	0.0	0.0	0.0
Enthalpy	J/kmol	-2.1605E+8	-3.0651E+8	-3.0210E+8	-1.9707E+8
Enthalpy	J/kg	-3.7181E+6	-5.1039E+6	-7.7351E+6	-2.5222E+6
Enthalpy	Watt	-9.0023E+6	-4.2570E+6	-8.3916E+6	-5.4743E+6
Entropy	J/kmol-K	-2.3899E+5	-4.2628E+5	-3.1015E+5	-3.2964E+5
Entropy	J/kg-K	-4112.819	-7098.273	-7941.350	-4218.865
Density	kmol/cum	16.09926	12.04800	21.49249	13.41157
Density	kg/cum	935.5141	723.5294	839.4020	1047.914
Average MW		58.10913	60.05389	39.05560	78.13504
Liq Vol 60F	cum/sec	2.23142E-3	1.05920E-3	1.31070E-3	1.97992E-3

Temperature profile plot:-

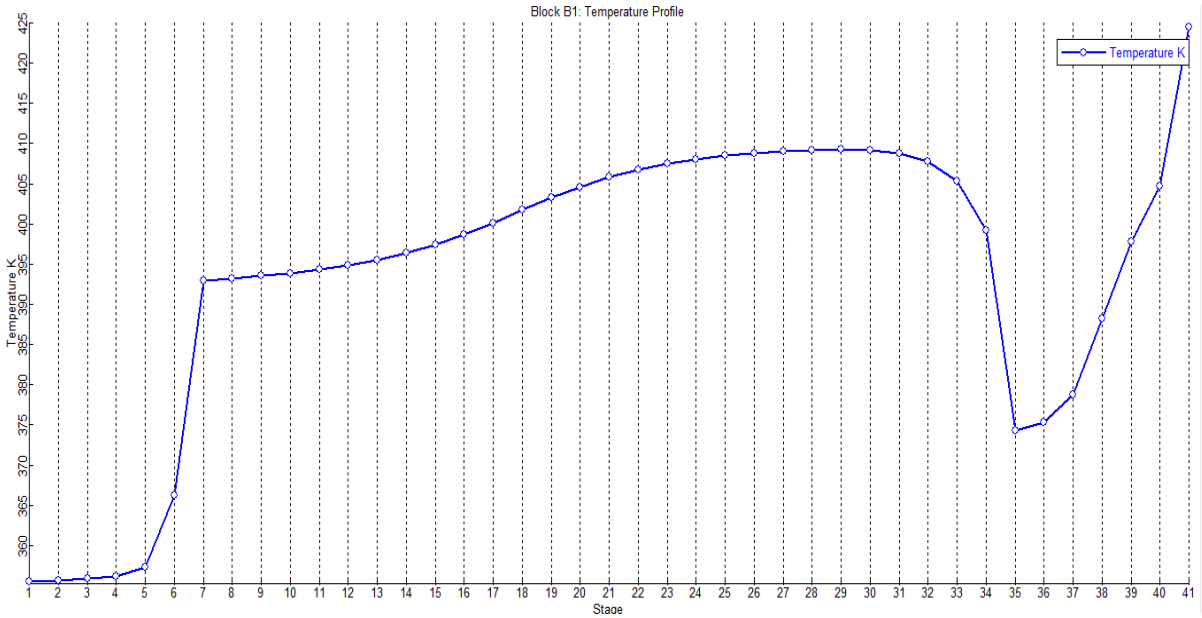

Fig-3.2 Temperature profile of column B1

Liquid Composition Profile:-

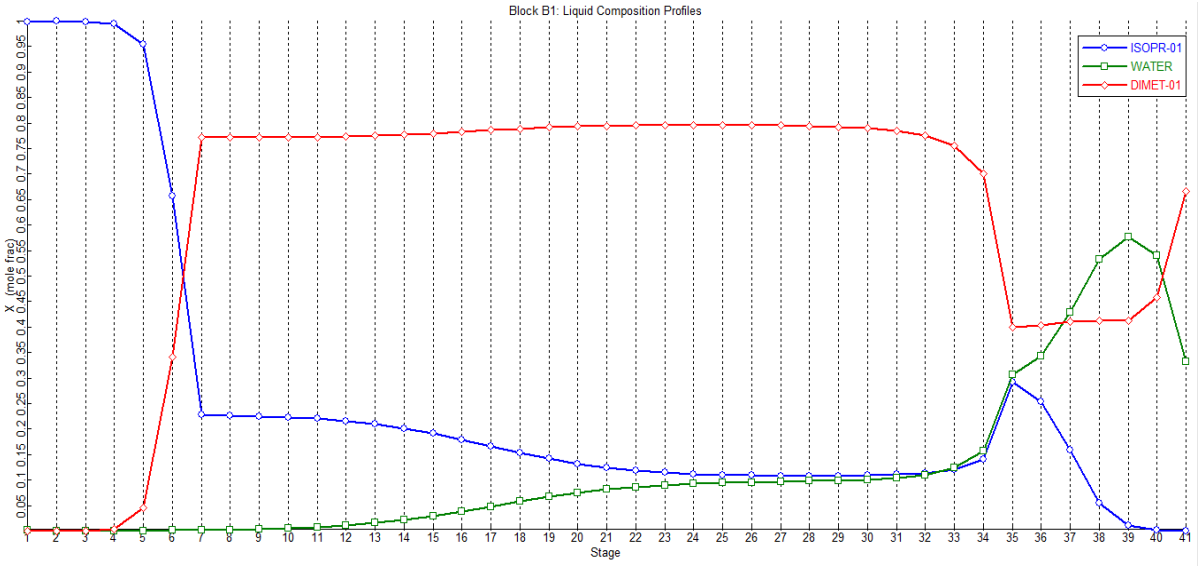

Fig-3.3 Liquid composition profile of column B1

Here we saw in extractive distillation using DMSO as entrainer we are getting 0.999 mole purity. WHY are we using extractive distillation in case of isopropanol water system??????

3.3 Simulating without Entrainer:-

If we simulate without using entrainer we are getting following results:-

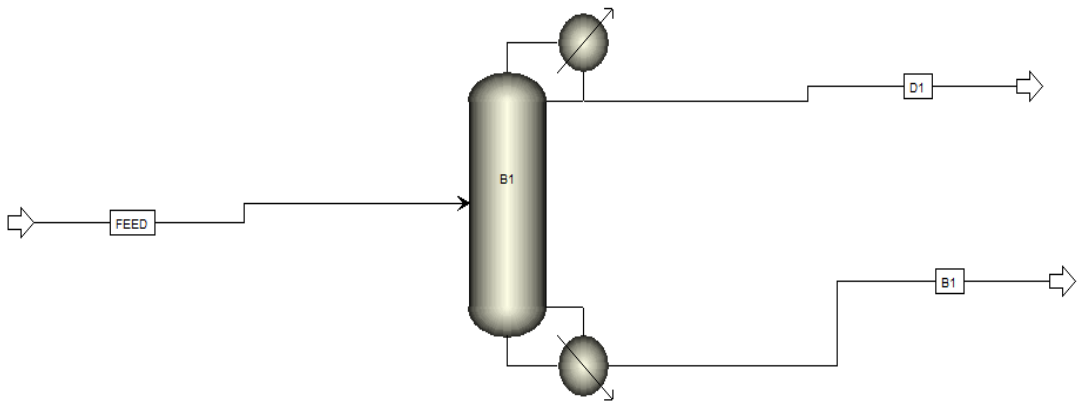

Fig-3.4 Setup used to simulate without entrainer

Table-3.3 Stream table of normal distillation

simple				
Stream ID		B1	D1	FEED
From		B1	B1	
To				B1
Phase		LIQUID	LIQUID	LIQUID
Substream: MIXED				
Mole Flow	kmol/sec			
ISOPR-01		7.45436E-3	6.43453E-3	.0138888
WATER		.0103234	3.56547E-3	.0138888
Total Flow	kmol/sec	.0177777	.0100000	.0277777
Total Flow	kg/sec	.6339557	.4509221	1.084878
Total Flow	cum/sec	8.20215E-4	5.98164E-4	1.29244E-3
Temperature	K	360.2507	353.5241	298.1500
Pressure	N/sqm	1.28885E+5	1.01325E+5	1.31723E+5
Vapor Frac		0.0	0.0	0.0

Liquid Frac		1.000000	1.000000	1.000000
Solid Frac		0.0	0.0	0.0
Enthalpy	J/kmol	-2.9143E+8	-2.9767E+8	-3.0210E+8
Enthalpy	J/kg	-8.1725E+6	-6.6014E+6	-7.7351E+6
Enthalpy	Watt	-5.1810E+6	-2.9767E+6	-8.3916E+6
Entropy	J/kmol-K	-2.6210E+5	-3.2534E+5	-3.1015E+5
Entropy	J/kg-K	-7350.002	-7214.892	-7941.350
Density	kmol/cum	21.67453	16.71782	21.49249
Density	kg/cum	772.9138	753.8435	839.4020
Average MW		35.66001	45.09221	39.05560
Liq Vol 60F	cum/sec	7.55259E-4	5.55444E-4	1.31070E-3

In this simulation we are getting negligible amount of IPA from top as distillate. Using design specs and vary we are getting error in converging.

```
<< Problem specifications modified 11:43:15 Mon Nov 3, 2014>>

->Processing input specifications ...

->Finished processing new specifications

->Calculations begin ...

    Block: B1        Model: RADFRAC

       Convergence iterations:
         OL    ML    IL      Err/Tol
          1     7    12       69.282
          2     2     3       32.198
          3     2     3       6.9478
          4     2     3      0.57968
  **   ERROR
       DESIGN SPEC IS NOT SATISFIED BECAUSE ONE OR MORE MANIPULATED
       VARIABLE IS AT ITS BOUND.

->Simulation calculations completed ...
```

Fig-3.5 Showing completion of simulation with error if design specs and vary is used

Now the Question arises why are we getting such result. Let's see the Txy plot of isopropanol-water system. From the plot it's clear that its making an minimum boiling mixture at mole fraction of IPA between 0.6 to 0.7.

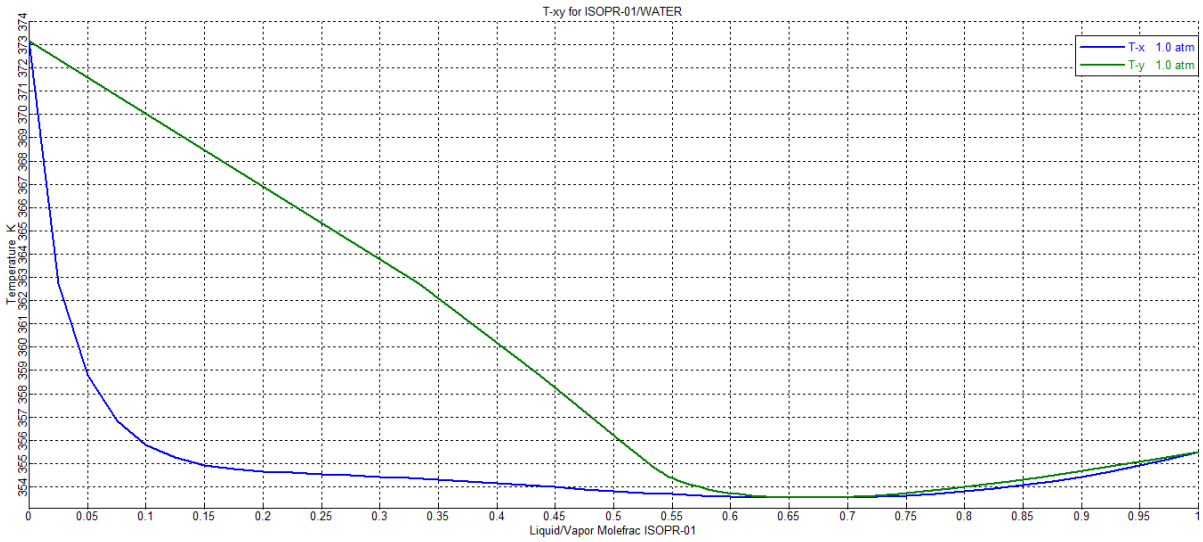

Fig-3.6Txy plot of IPA-water

Boiling point of water= 100^{o}C

Boiling Point of IPA= 82.6^{o}C

Boiling Point of azeotrope= 80.6^{o}C

Analysis:-

From the above simulation of isopropanol water mixture using DMSO as entrainer shown in fig 4.1, we are getting 0.99999 mole fraction of IPA from top bottom product has a mixture of water and DMSO having 0.666 and 0.333 mole fraction respectively. But we cannot loose DMSO in every run so we need another distillation column where DMSO and water can be separated with their maximum purity. Setup is as shown below:-

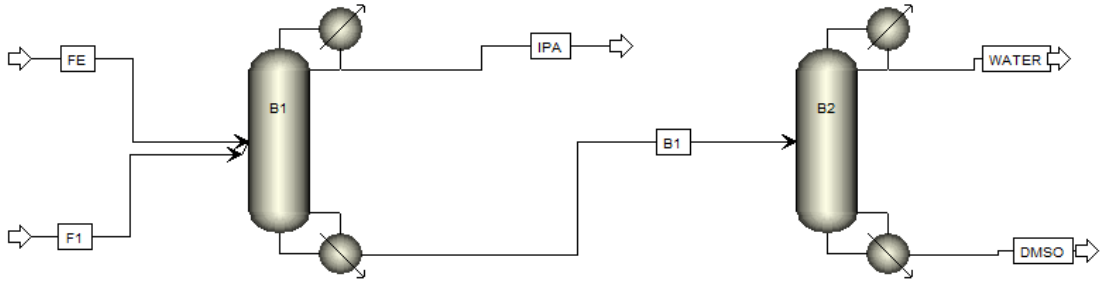

Fig-3.7: Complete setup to separate the three components

Block B2 is the additional column used to separate DMSO and water mixture from the bottom of block B1 obtained. Conditions used are as follows:-

No of stages – 18

Feed in 9^{th}stage (Above stage)

Condenser pressure- 1atm.

Results so obtained are as given in the table below

Table- 3.4 Stream table of extractive distillation column along with recovery column

final year pro							
Stream ID		B1	DMSO	F1	FE	IPA	WATER
From		B1	B2			B1	B2
To		B2		B1	B1		
Phase		LIQUID	LIQUID	LIQUID	LIQUID	LIQUID	LIQUID
Substream: MIXED							
Mole Flow	kmol/sec						
ISOPR-01		3.53665E-7	3.4341E-17	.0138888	0.0	.0138885	3.53665E-7
WATER		.0138749	2.88480E-6	.0138888	0.0	1.39009E-5	.0138721
DIMET-01		.0277777	.0277613	0.0	.0277777	1.55693E-9	1.64320E-5
Mole Frac							
ISOPR-01		8.49072E-6	1.2369E-15	.5000000	0.0	.9990000	2.54639E-5
WATER		.3331080	1.03903E-4	.5000000	0.0	9.99888E-4	.9987914
DIMET-01		.6668835	.9998961	0.0	1.000000	1.11990E-7	1.18311E-3
Total Flow	kmol/sec	.0416531	.0277642	.0277777	.0277777	.0139024	.0138888
Total Flow	kg/sec	2.420401	2.169186	1.084878	2.170418	.8348949	.2512150
Total Flow	cum/sec	2.58738E-3	2.34937E-3	1.29244E-3	2.07118E-3	1.15392E-3	2.73724E-4
Temperature	K	424.6471	468.1491	298.1500	345.1500	355.4828	373.1816
Pressure	N/sqm	1.28885E+5	1.13038E+5	1.31723E+5	1.11458E+5	1.01325E+5	1.01325E+5
Vapor Frac		0.0	0.0	0.0	0.0	0.0	0.0
Liquid Frac		1.000000	1.000000	1.000000	1.000000	1.000000	1.000000
Solid Frac		0.0	0.0	0.0	0.0	0.0	0.0
Enthalpy	J/kmol	-2.1601E+8	-1.7627E+8	-3.0210E+8	-1.9707E+8	-3.0651E+8	-2.7999E+8
Enthalpy	J/kg	-3.7173E+6	-2.2562E+6	-7.7351E+6	-2.5222E+6	-5.1039E+6	-1.5480E+7
Enthalpy	Watt	-8.9974E+6	-4.8940E+6	-8.3916E+6	-5.4743E+6	-4.2612E+6	-3.8888E+6
Entropy	J/kmol-K	-2.3891E+5	-2.7878E+5	-3.1015E+5	-3.2964E+5	-4.2628E+5	-1.4626E+5
Entropy	J/kg-K	-4111.516	-3568.194	-7941.350	-4218.865	-7098.278	-8086.388
Density	kmol/cum	16.09859	11.81773	21.49249	13.41157	12.04801	50.74049
Density	kg/cum	935.4652	923.3048	839.4020	1047.914	723.5292	917.7676
Average MW		58.10851	78.12879	39.05560	78.13504	60.05385	18.08748
Liq Vol 60F	cum/sec	2.23039E-3	1.97880E-3	1.31070E-3	1.97992E-3	1.06023E-3	2.51590E-4

Optimum result is obtained with purity of IPA, water and DMSO is found to be 0.9999. But here we are using 2 columns to obtain the above result.

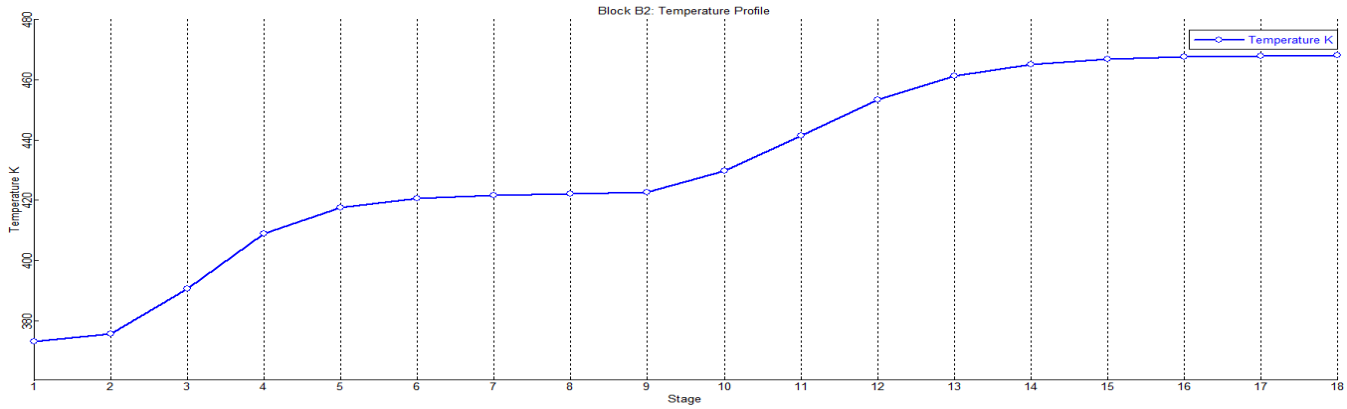

Fig-3.8 Temperature profile of column B2

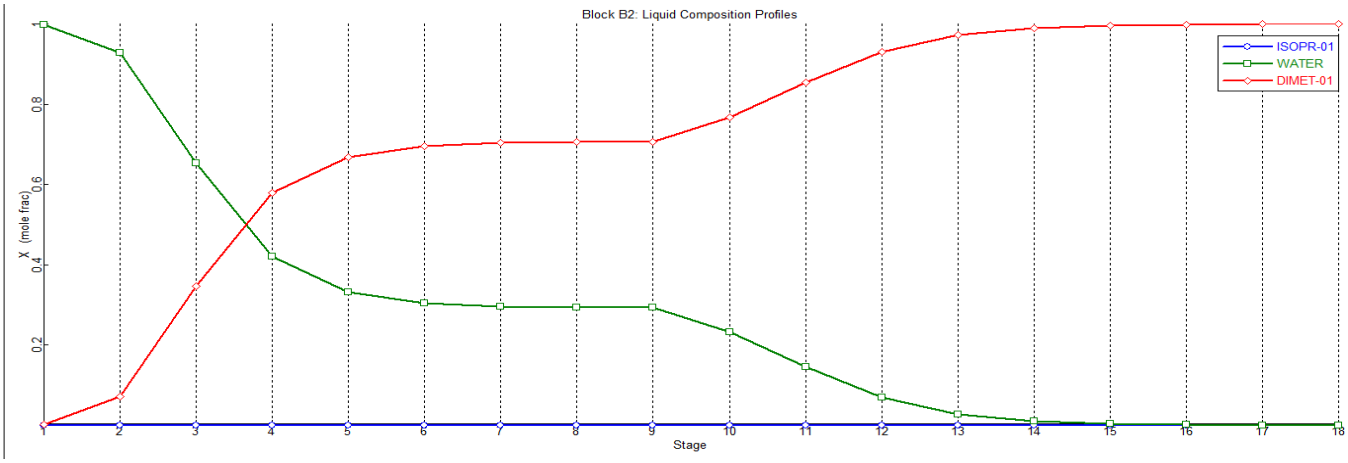

Fig-3.9 Liquid composition profile of column B2

Analysis:-

As discussed earlier we can't lose DMSO in every run. Bottom product from column B1 contains 0.66666 DMSO and 0.33333 water. So simulating with another column with 18 plates where 9th plate is feed stage we are getting 0.998 water and 0.999 DMSO. Hence complete separation of DMSO, water and IPA is completed. This is a conventional method followed in major industries. In temperature profile there is a normal rise in temperature down the column. More volatile component water as compared to DMSO is obtained from the top as shown in fig 4.7.

Nowadays energy conservation has been a key point to progress in production line. Every step are taken to save energy as far as possible. In this method two columns are used. In Divided wall distillation column the above result is achieved using single column.

CHAPTER 4

DIVIDED WALL DITILLATION COLUMN

In recent years, more energy efficient distillation columns have been designed such as the Petlyuk column and divided wall column (DWC) which have greatly reduced the reboiler heat duty for a given separation.

The DWC is a configuration of two basic thermally coupled distillation sections put into one section by creating a vertical divider isolating the centre in two sections that act as the pre or post-fractionator and the main column. Thermodynamically, a DWC can be considered as an equivalent of the Petlyuk column [9]. The only difference between a DWC and a Petlyuk column lies in the stage/plate at which the liquid splitting and vapour return take place. The DWC has compact design; require less space and has higher vitality productivity when compared with a traditional distillation column. Here, an azeotropic mixture or mixture having multiple components can be separated in one column. In case of an azeotropic mixture, an entrainer is used to separate the mixture where both components along with the entrainer are separated individually from a single column. This is a new method taking shape now-a-days accepted widely across industries. DWC is widely used in linear alkyl benzene (LAB) forming industries. In LAB production, when the output is about 2.6 MT/yr, DWC saves 9% of total energy when compared with conventional distillation columns. A sum of around $12.8 million is saved per year on installing DWC rather than using conventional distillation column [10].

A model distillation column is shown below:

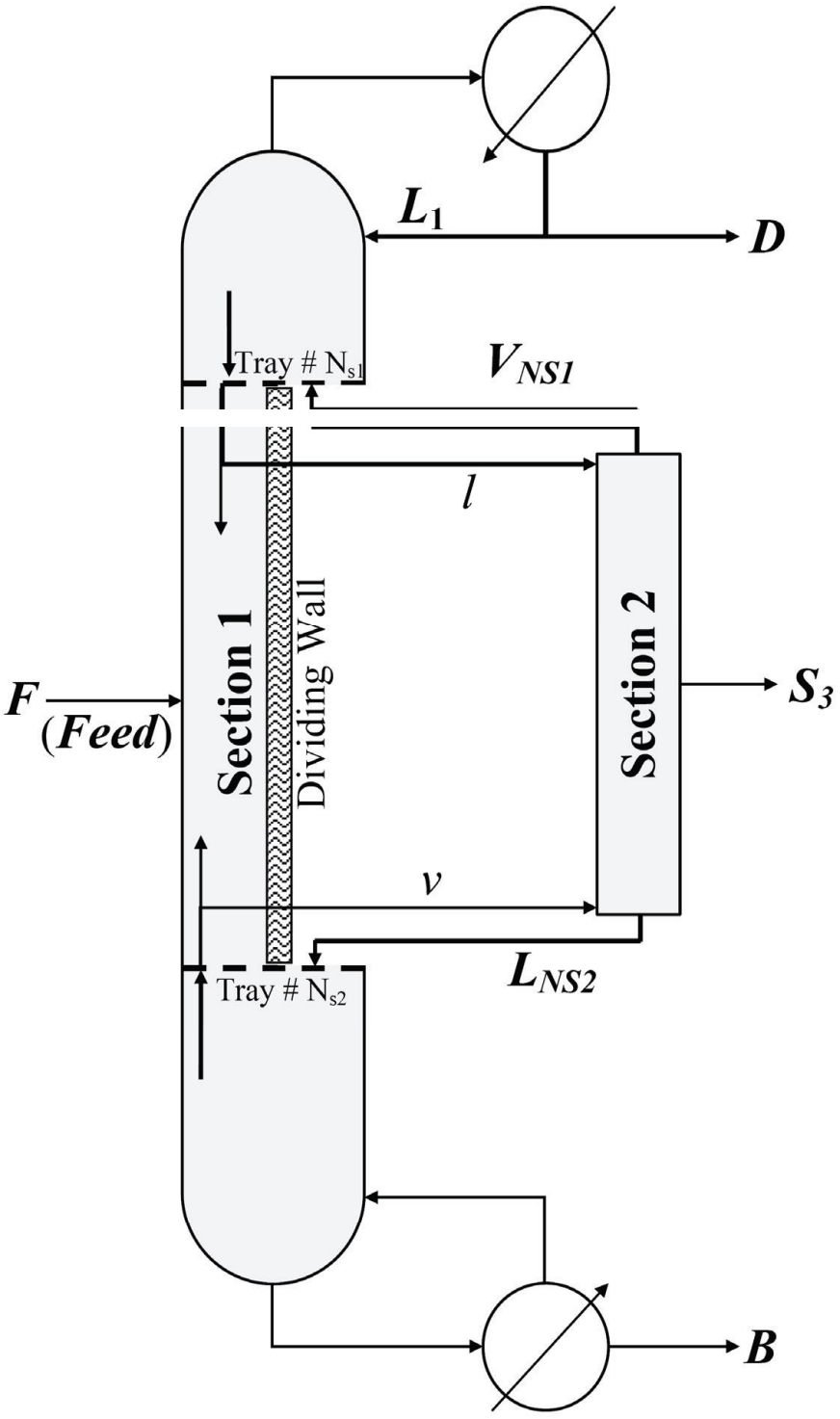

Fig 4.1 Divided wall distillation column[10]

We will try to simulate the IPA and water mixture using DMSO as entrainer as performed above in conventional distillation column and try to optimize it. Main components are IPA and DMSO. We have to obtain its purity as high as possible. Simulation is done in ASPEN

PLUS 11.1 and V8.04 software. Direct distillation column is not available in column section. Hence we are using 4 columns which will be analogous to DWC. Those four columns include two absorption columns, one rectifying column and one stripping column.One mixer and one splitter is used.

4.1 Simulation performed

In this section we are splitting the divided wall distillation column into four columns. Two absorption column namely MAIN and SIDE columns. One rectifying column named as ENRICH and one stripping column named as STRIP. Along with that two mixers and one splitter is used namely MIX1 , MIX2 and SPLIT1 respectively. Following arrangements were done as shown in diagram below:-

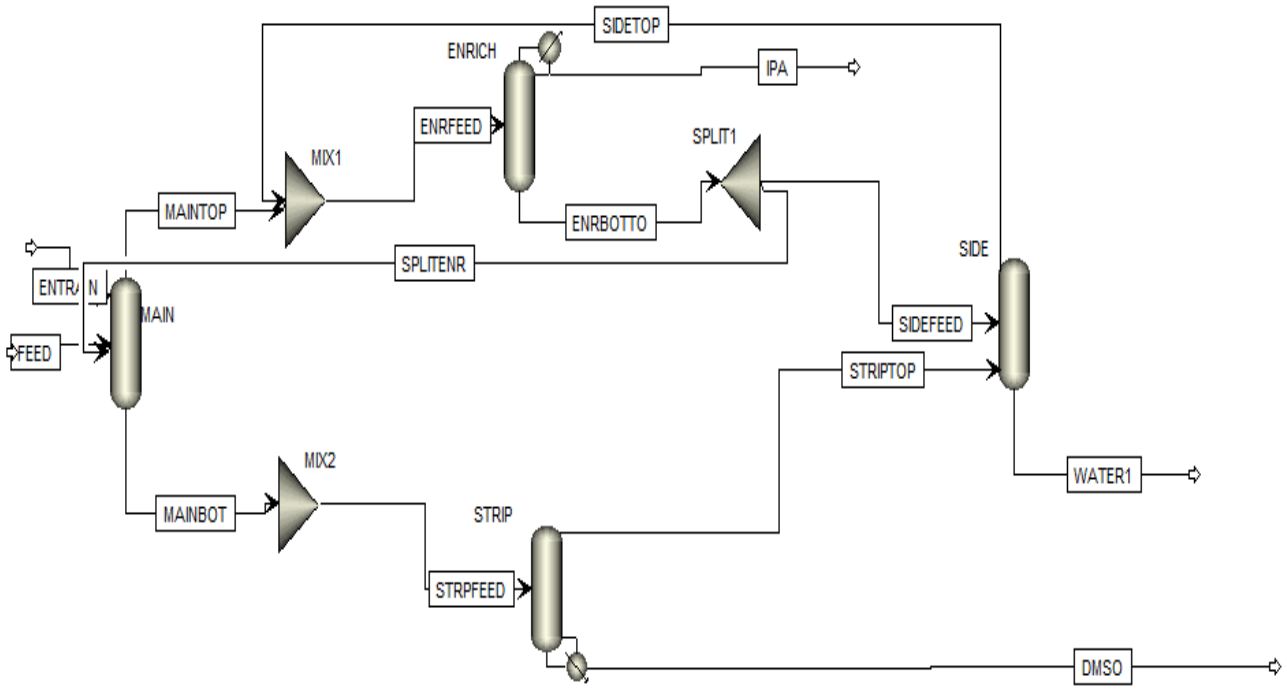

Fig 4.2 Arrangement used in simulation analogous to DWC

Conditions here used are given as follows:-

MAIN COLUMN

Feed line is given to 15th plate to MAIN column which consist of 0.5 mole fraction of IPA and water respectively. Entrainer used is DMSO which is given to MAIN column through ENTRAIN line both feed and entrainer given as 100kmol/hr. Number of plates taken as 29. Feed pressure is taken as 1.3 atm where as entrainer line pressure is taken as 1.1 atm. Condenser pressure is 1 atm. Here condenser is none and reboiler used is kettle.

ENRICH COLUMN

Here column taken is a rectifying column. All the top products from MAIN column and SIDE column moves into it ENRICH column at 9th stage (onstage). Number of stages used is 9. Further condenser pressure is 1 atm. Reboiler is none. Distillate rate is 50 kmol/hr.

SPLIT1

This is a major section of the process as alteration at this point i.e split ratio results in change in concentration of final product. Split ratio is taken to be 0.9 to SIDE column and 0.1 back to main column. Reducing the value from 0.9 results in decrease in concentration of water from SIDE column. Further increase in split ratio from 0.9 results in decrease in IPA concentration.

SIDE COLUMN

Column here taken is an absorption column. Bottom product from ENRICH column is splited in SPLIT1 out of which 0.9 split ratio is sent into SIDE column in 1st stage(onstage) . It has total 29 stages. Condenser pressure maintained is 1atm. Condenser and reboiler is taken as none. Water is taken out from the bottom of the column. Top product from STRIP column is fed into 29th stage (onstage) and no splitter is used here.

STRIP COLUMN

This column is used for stripping purpose. Stripping column is used as almost all water along with DMSO is move down to this column and is fed in 1stplate (onstage). Condenser here is none and reboiler is kettle. Boil up ratio is used. It's taken as 0.9. Decrease in boilup ratio lead to decrease in DMSO concentration as product whereas further increase in boilup ratio results in no change in DMSO concentration. Hence for optimum condition 0.9 is taken.

MIX1 , MIX2, SPLIT1

Mixers and splitters are used to amplify the result obtained here as in real DWC proper mixing and splitting is performed. From the above explanations MIX1 and SPLIT1 function is known. MIX2 is not necessary but taken only for proper mixing it can also be removed and required results can be obtained.

Performing simulations with the above conditions we are getting the results as shown below:-

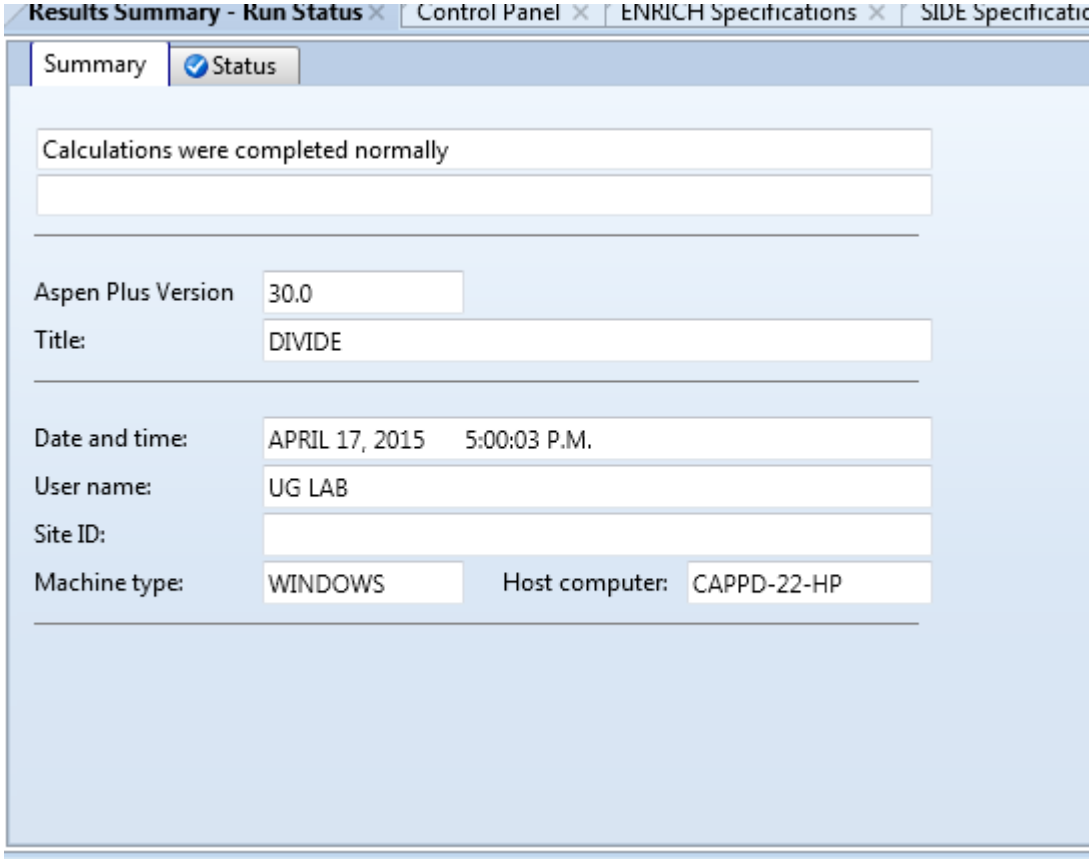

Fig4.3Status of simulation run

Table 4.1Steam table of divided wall distillation column

divide															
Stream ID		DMSO	ENRBOTTO	ENRFEED	ENTRAIN	FEED	IPA	MAINBOT	MAINTOP	SIDEFEED	SIDETOP	SPLITENR	STRIPTOP	STRPFEED	WATER1
From		STRIP	ENRICH	MIX1			ENRICH	MAIN	MAIN	SPLIT1	SIDE	SPLIT1	STRIP	MIX2	SIDE
To			SPLIT1	ENRICH	MAIN	MAIN		MIX2	MIX1	SIDE	MIX1	MAIN	SIDE	STRIP	
Phase		LIQUID	LIQUID	MIXED	LIQUID	LIQUID	LIQUID	LIQUID	VAPOR	LIQUID	VAPOR	LIQUID	VAPOR	MIXED	LIQUID
Substream: MIXED															
Mole Flow	kmol/hr														
WATER		.5571935	1.102486	1.824846	0.0	50.00000	.7223606	49.22321	.8870343	.9922372	.9377776	.1102486	48.66602	49.22321	48.72048
ISOPR-01		2.2490E-11	104.5604	123.8380	0.0	50.00000	19.27764	6.213229	54.24281	94.10434	69.59496	10.45604	6.213229	6.213229	30.72260
DIMET-01		86.89132	7.253719	7.253719	100.0000	0.0	9.8237E-13	93.59936	7.126011	6.528347	.1276909	.7253719	6.708039	93.59936	13.10870
Mole Frac															
WATER		6.37168E-3	9.76372E-3	.0137292	0.0	.5000000	.0361180	.3302778	.0142482	9.76372E-3	.0132716	9.76372E-3	.7901959	.3302778	.5264132
ISOPR-01		2.5718E-13	.9259967	.9316973	0.0	.5000000	.9638820	.0416895	.8712885	.9259967	.9849213	.9259967	.1008849	.0416895	.3319504
DIMET-01		.9936283	.0642396	.0545734	1.000000	0.0	4.9118E-14	.6280327	.1144633	.0642396	1.80711E-3	.0642396	.1089192	.6280327	.1416363
Total Flow	kmol/hr	87.44851	112.9166	132.9166	100.0000	100.0000	20.00000	149.0358	62.25585	101.6249	70.66043	11.29166	61.58729	149.0358	92.55178
Total Flow	kg/hr	6799.295	6870.283	8041.804	7813.504	3905.560	1171.521	8573.550	3832.543	6183.255	4209.245	687.0283	1774.255	8573.550	3748.265
Total Flow	l/min	121.8998	154.2020	68650.95	127.4167	79.33664	26.93293	151.1016	34017.47	138.7818	34468.41	15.42020	34474.00	151.1016	74.64223
Temperature	C	189.4093	83.54006	108.7768	100.0000	40.00000	81.62396	127.1168	126.3920	83.54006	83.53564	83.54006	136.1495	127.1168	84.11712
Pressure	bar	1.013250	1.013250	1.013250	1.114575	1.300000	1.013250	1.013250	1.013250	1.013250	1.013250	1.013250	1.013250	1.013250	1.013250
Vapor Frac		0.0	0.0	.9888164	0.0	0.0	0.0	0.0	1.000000	0.0	1.000000	0.0	1.000000	2.2924E-10	0.0
Liquid Frac		1.000000	1.000000	.0111835	1.000000	1.000000	1.000000	1.000000	0.0	1.000000	0.0	1.000000	0.0	1.000000	1.000000
Solid Frac		0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Enthalpy	cal/mol	-42662.80	-71501.63	-61469.93	-45977.62	-71763.89	-73146.84	-53453.64	-59152.17	-71501.63	-63512.16	-71501.63	-54838.68	-53453.64	-66513.22
Enthalpy	cal/gm	-548.7037	-1175.166	-1015.988	-588.4379	-1837.480	-1248.750	-929.1957	-960.8683	-1175.166	-1066.176	-1175.166	-1903.540	-929.1957	-1642.338
Enthalpy	cal/sec	-1.0363E+6	-2.2427E+6	-2.2695E+6	-1.2772E+6	-1.9934E+6	-4.0637E+5	-2.2129E+6	-1.0229E+6	-2.0184E+6	-1.2466E+6	-2.2427E+5	-9.3816E+5	-2.2129E+6	-1.7100E+6
Entropy	cal/mol-K	-67.16177	-97.96936	-69.47997	-75.67671	-72.08986	-98.30786	-59.84123	-66.34460	-97.96936	-72.46702	-97.96936	-17.31563	-59.84123	-62.15859
Entropy	cal/gm-K	-.8637950	-1.610176	-1.148379	-.9685375	-1.845826	-1.678294	-1.040233	-1.077702	-1.610176	-1.216501	-1.610176	-.6010538	-1.040233	-1.534814
Density	mol/cc	.0119563	.0122044	3.22687E-5	.0130804	.0210075	.0123764	.0164388	3.05019E-5	.0122044	3.41668E-5	.0122044	2.97748E-5	.0164388	.0206656
Density	gm/cc	.9296292	.7425632	1.95234E-3	1.022041	.8204616	.7249619	.9456716	1.87773E-3	.7425632	2.03532E-3	.7425632	8.57774E-4	.9456715	.8369402
Average MW		77.75198	60.84388	60.50264	78.13504	39.05560	58.57605	57.52678	61.56116	60.84388	59.57004	60.84388	28.80878	57.52678	40.49911
Liq Vol 60F	l/min	103.3902	141.9506	166.6892	118.7950	78.64217	24.73866	133.9026	77.72959	127.7555	88.95929	14.19506	30.51247	133.9026	69.30868

Temperature Profiles:-

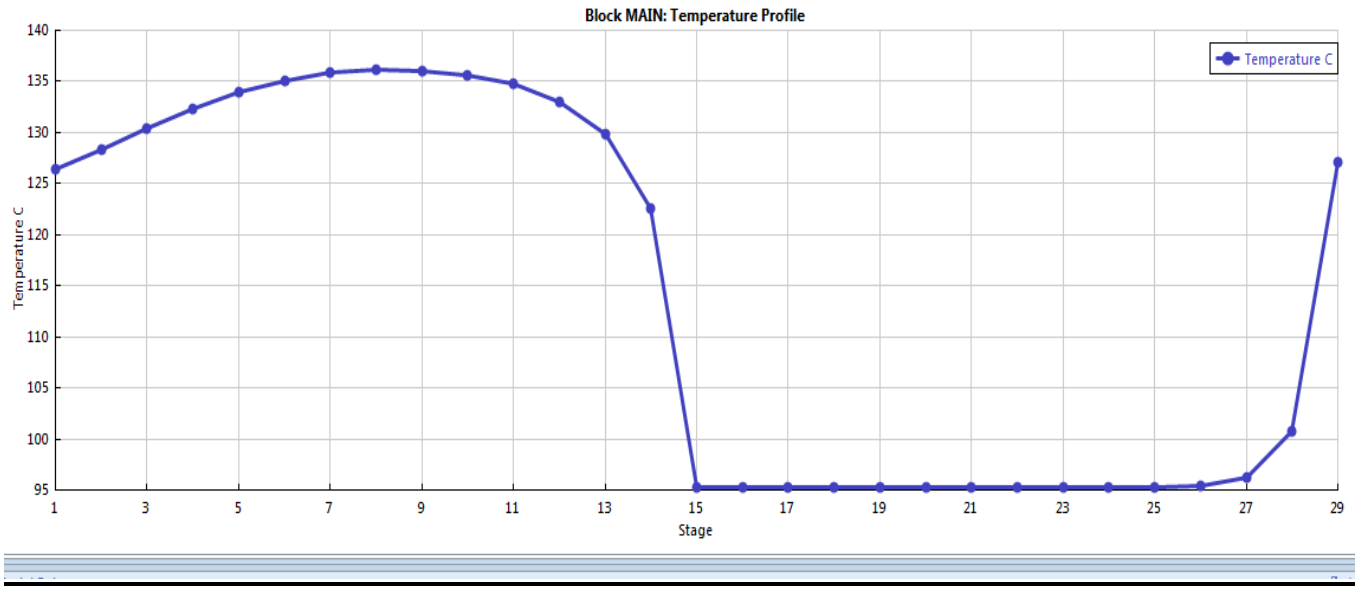

Fig 4.4Temperature Profile of column MAIN

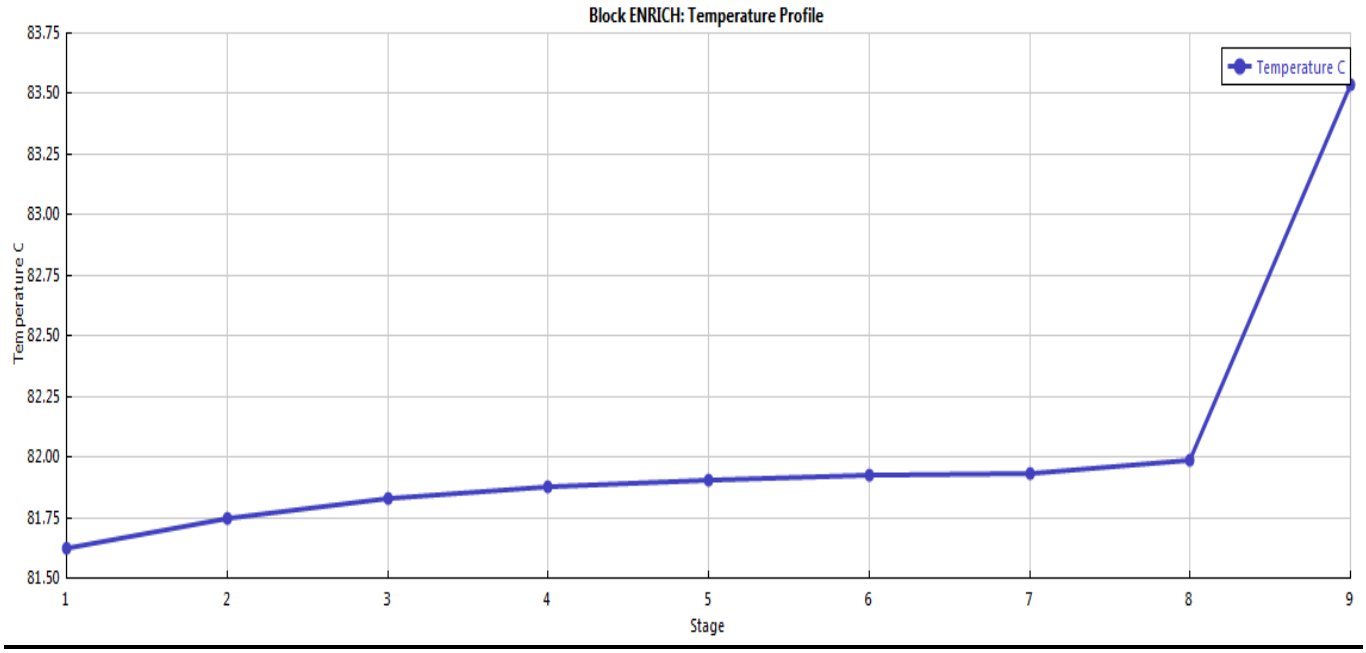

Fig 4.5Temperature Profile of column ENRICH

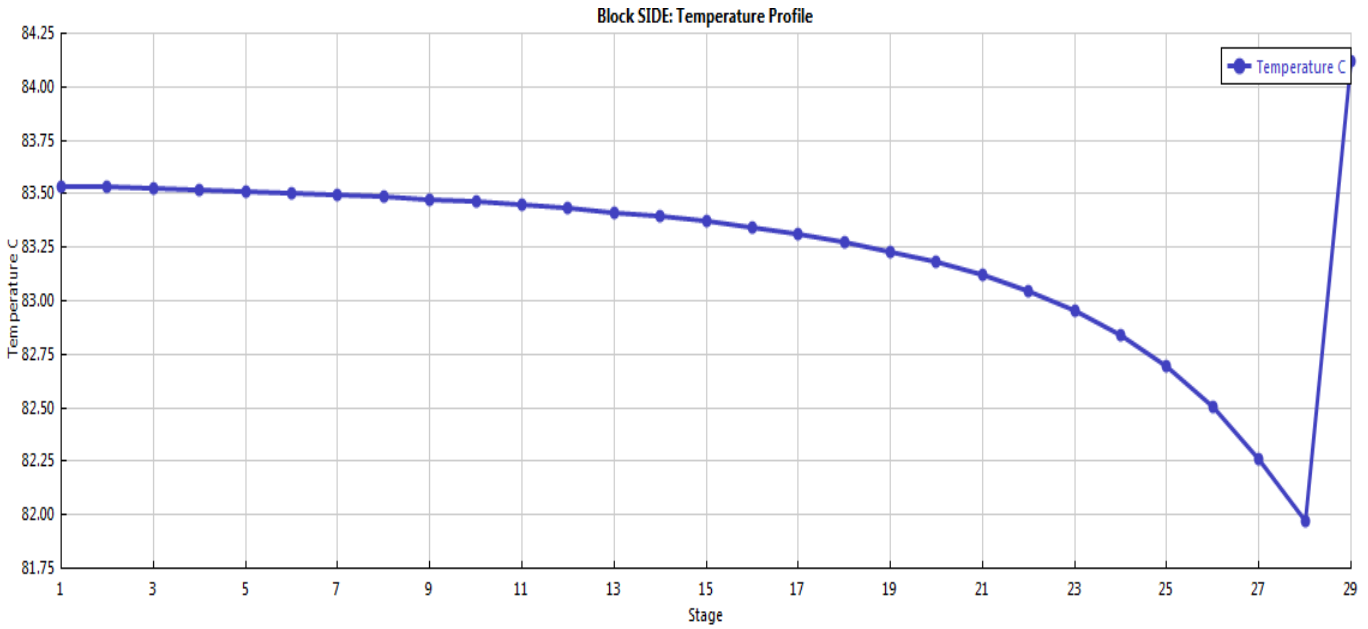

Fig 4.6Temperature Profile of column SIDE

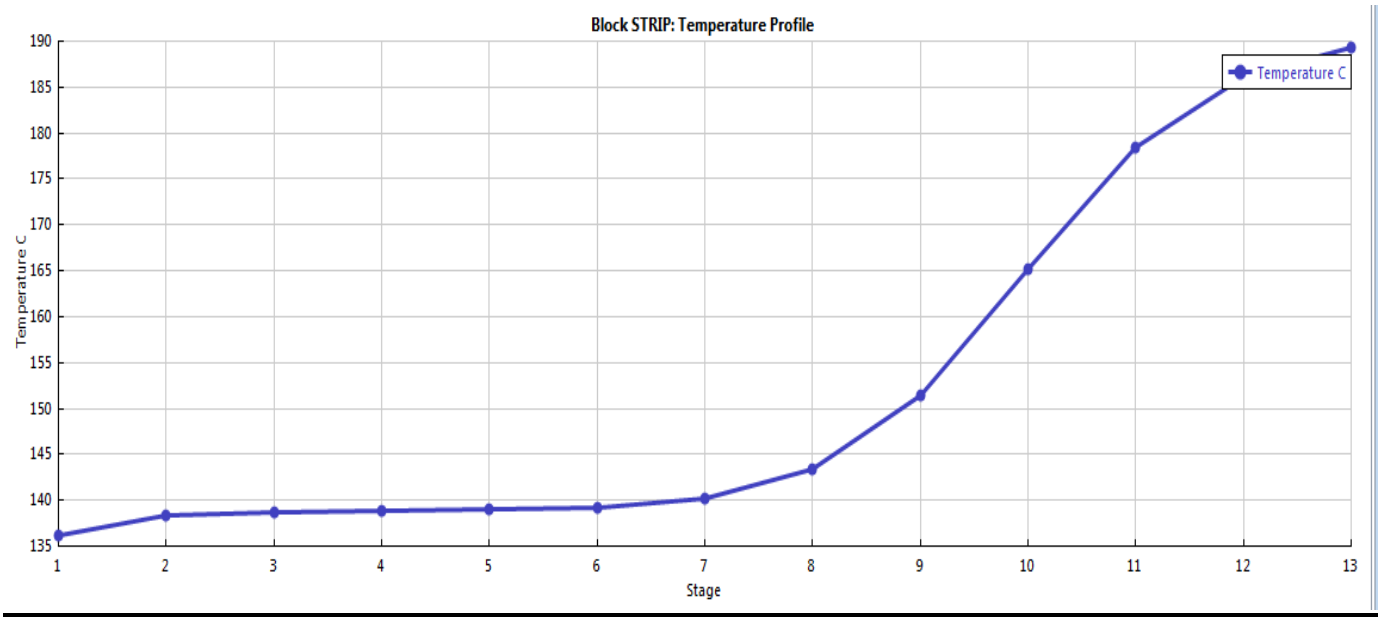

Fig 4.7Temperature Profile of column STRIP

Liquid composition profiles:-

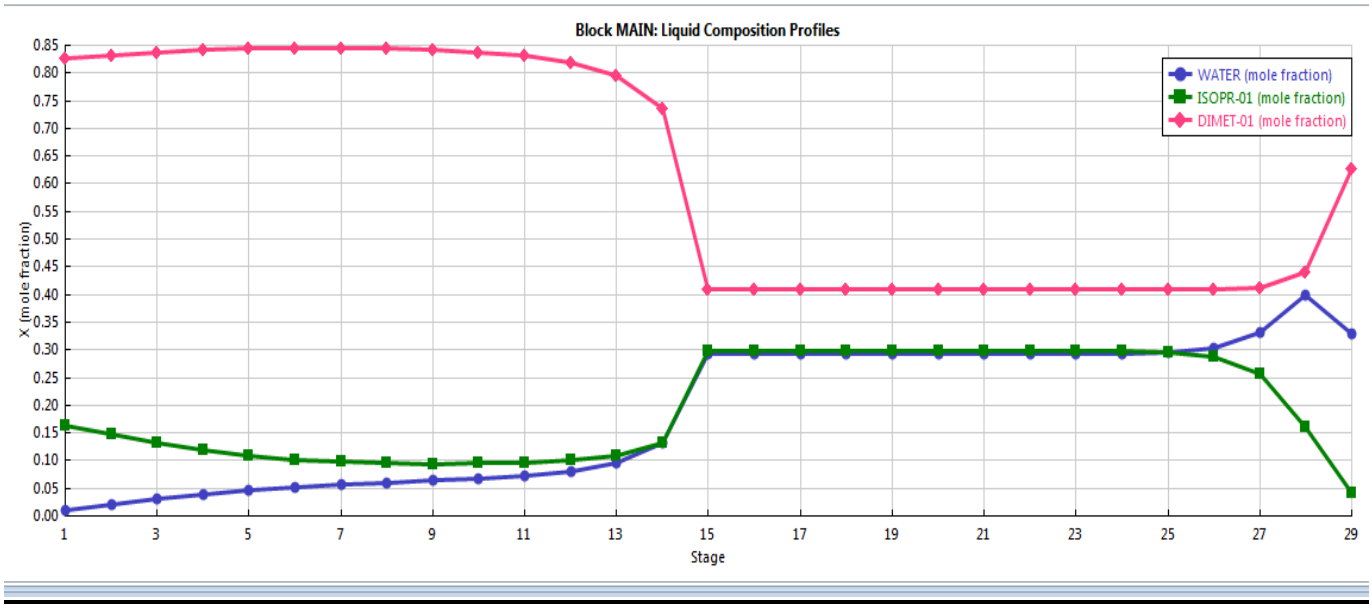

Fig 4.8Liquid composition profile of column MAIN

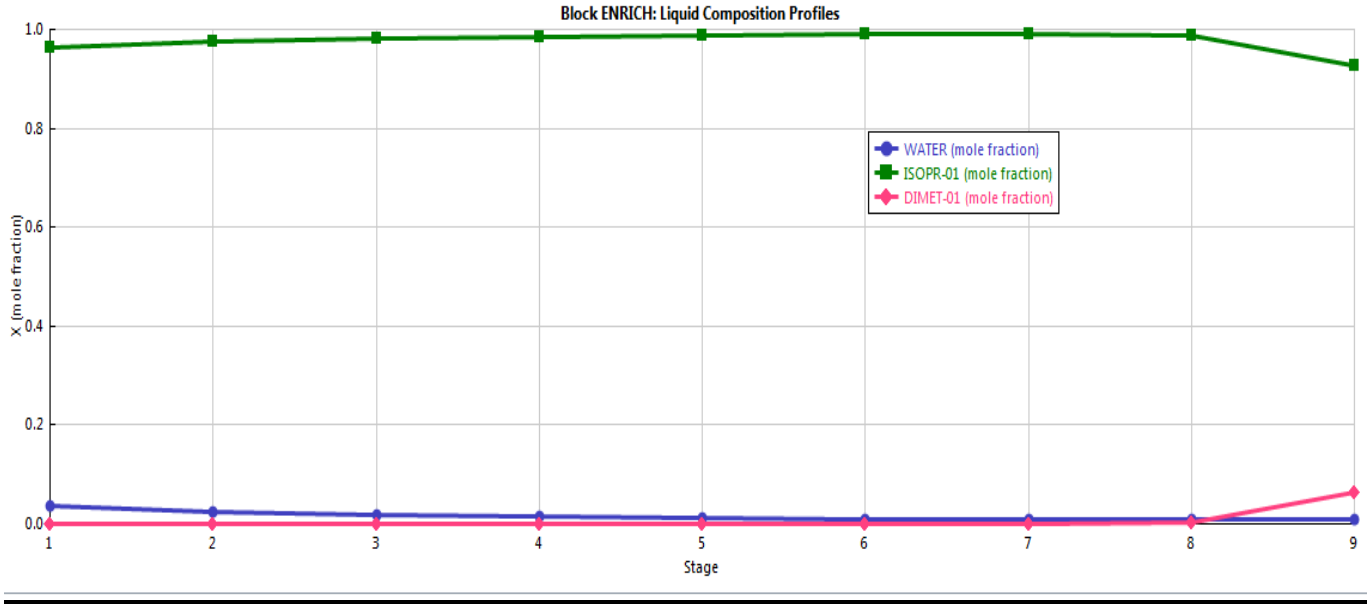

Fig 4.9Liquid composition profile of column ENRICH

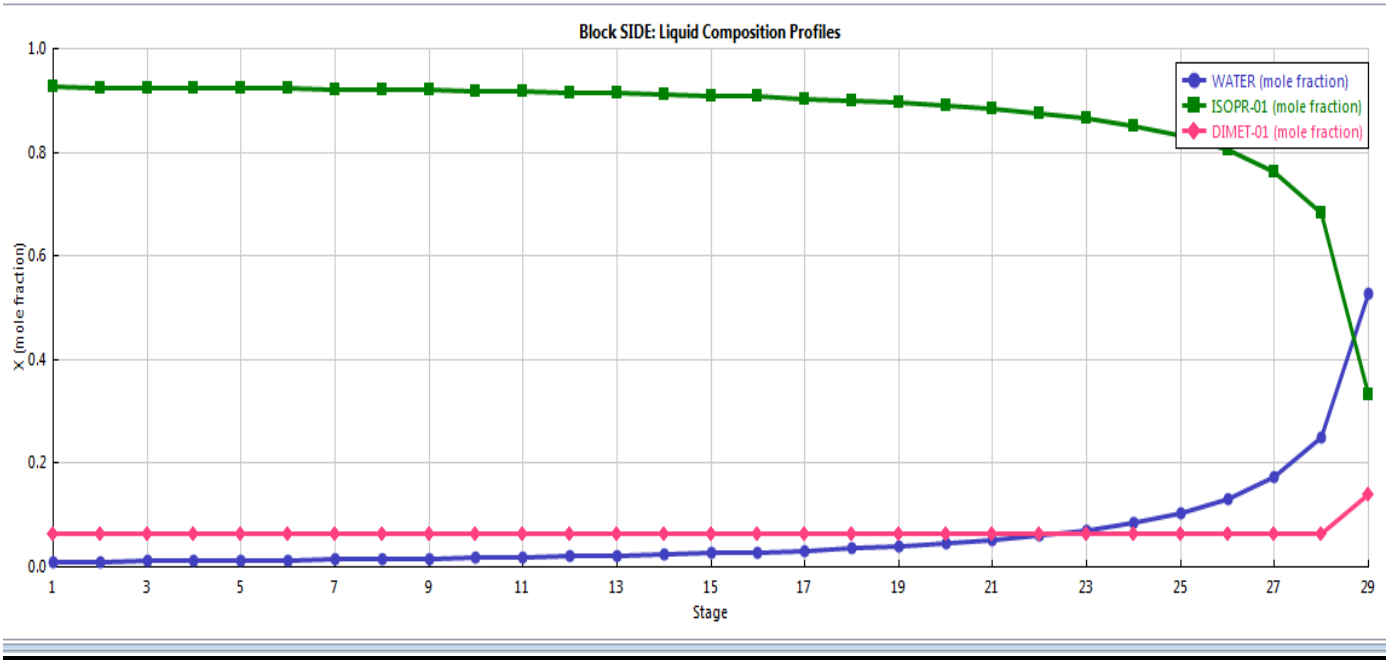

Fig 4.10Liquid composition profile of column SIDE

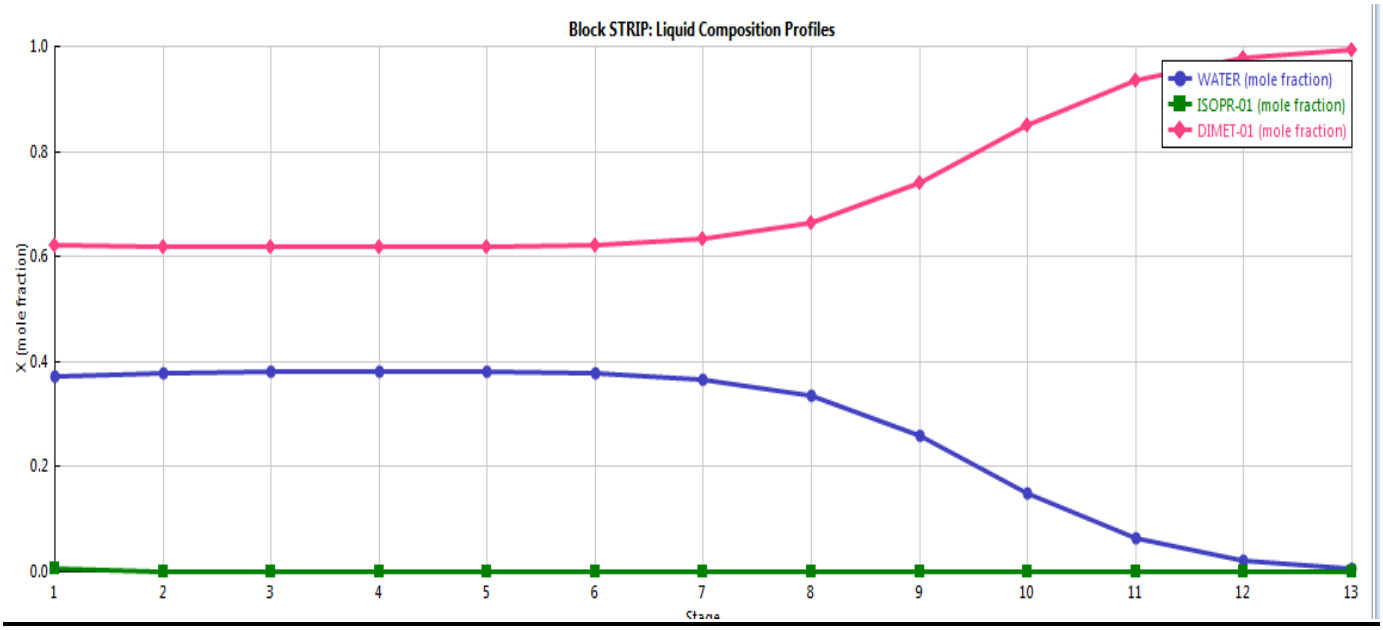

Fig 4.11Liquid composition profile of column STRIP

4.2 Analysis

In this entire setup model used is RADFRAC distillation column. This is the most suitable option available since it provides flexibility in reboiler and condenser conditions. In this model we can take condenser and reboiler as none. In addition to that vaporization or murphee efficiency can be specified in stage or component basis. This model is user friendly in various options and provides a wide range of flexibility. Heat curves of reboiler and condenser can easily be plotted using this model.The liquid split factor in the dividing wall column decides how much quantity of the liquid will enter the SIDE column from the rectifying section. Liquid from the bottom of rectifying section is rich in water and DMSO component which are to be separated in SIDE column hence major portion of liquid is sent to SIDE column (0.9).

A splitter was also used in between stripper column and SIDE column but water mole fraction was coming to be 0.0104 which is very minimal. At that time water was withdrawn from 28^{th} plate. On removing the splitter and sending the entire top product of stripping column into SIDE column gives water mole fraction to be 0.555 which is nearly equal to feed proportion of 0.5 hence can be used as recycled product. Water line constitutes 0.52 water, 0.33 IPA and 0.15 DMSO. Upon modification we are getting 0.9638 IPA from top 0.9936 from bottom DMSO which are the major components in this entire simulation. In stripping section boil-up ratio is taken to be 0.9.

CHAPTER 5

CONCLUSIONS AND FUTURE WORKS

We have successfully simulated an extractive as well as divided wall distillation system using Aspen-plus for separating isopropanol-water mixture using dimethyl sulfoxide (DMSO) as an entrainer. The extractive distillation system required 2 distillation column assemblies for achieving desired degrees of purity whereas in divided wall system, the target values were achieved in a single assembly, reducing the requirements of additional auxiliaries. The salient outcomes of this work are outlined below:

(a) The total number of theoretical plates required for achieving 99% and higher mole purity w.r.t isopropanol (IPA) was found to be 41 (out of which 39 were trays and 2 were for reboiler and condenser with reflux drum). The optimum feed and entrainer plate was located at 35^{th} and 7^{th} stage respectively (counted from top down). The values of the manipulated variables *viz*. distillate flow rate and reflux ratio were *ca*. 50 kmol/h and 0.6895 respectively for achieving 99% and higher mole purity of the distillate product (isopropanol).

(b) A significant percentage of DMSO remained in the bottom product of the first column of the extractive system which was successfully recovered (>99%) as a bottom product from the auxiliary column of the assembly.

(c) Since this process involved two distillation columns, a relatively more contemporary and modern distillation mechanism was resorted to *viz*. divided wall distillation process. Improvisations were made in the flow sheet to mimic divided wall distillation mechanism as Aspen-plus don't have such specific simulator. The results were comparable to what was achieved in extractive distillation process. The simulation results predicted mole purities of 0.9638 (isopropanol), 0.993 (DMSO) and 0.52 (water) respectively.

The future scope of this work can be the following:

(a) Detailed investigation of similar azeotropic mixtures using extractive distillation vis-à-vis divided wall distillation systems.

(b) Studies on performances of several other entrainers on isopropanol-water mixture.

(c) Dynamic simulation study of isopropanol-water system by introducing controlling mechanisms.

REFERENCES

[1] Segovia-Hernandez. J.G.Extractive Dividing Wall Column: Design and Optimization. Ind.Eng.Chem.Res.,49,3672-3688(2010)

[2]Luyben,William L, I-LungChien. Design and control of distillation systems for separating azeotropes.1-94(2010)

[3]Stupin W. J., Lockhart, F. J. Thermally coupled distillation-a case history.Chem. Eng. Program, 68, 71-72(1972)

[4]Serra, M., Espuna, A. &Puigjaner, L. Control and optimization of the divided wall column. Chemical Engineering and Processing, 38, 549-562 (1999)

[5]Hernandez S. ,Gabriel S. H. Thermodynamically equivalent distillation schemes to the Petlyuk column for ternary mixtures. Energy, 31, 2176-2183 (2006)

[6]Asprion N. ,Kaibel G. Dividing wall columns: Fundamentals and recent Advances. Chemical Engineering and Processing: Process Intensification, 49, 139-146 (2010)

[7]VanDiggelen R.C., Kiss A.A., Heemink A.W. Comparison of Control Strategies for Dividing-Wall Columns. Industrial& Engineering Chemistry Research, 49, 288-307 (2010)

[8]Hiller C., Buck C., Ehlers C., Fieg G. Non-equilibrium stage modelling of Dividing wall columns and experimental validation. Heat & Mass Transfer, 46, 1209–1220 (2010)

[9] PetlyukPlatonovV. M., SlavinskiiD. M., Int. Chem. Eng. J.5.555–561(1965)

[10]Sangal V.K., KumarVineet, MishraIndra Mani.Chemical Industry & Chemical EngineerinGQuarterly.19 (1) 107−119 (2013)